

PATENT COOPERATION TREATY

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PCT

Oy JALU ANT-WUORINEN AI

To:

OY JALO ANT-WUORINEN AB
Iso Roobertinkatu 4-6 A
FIN-00120 Helsinki
Finland

NOTIFICATION OF TRANSMITTAL OF
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IMPORTANT NOTIFICATION

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|---|--|--|

Applicant
Steris Europe inc. Suomen Sivuliike
et al

1. The applicant is hereby notified that this International Preliminary Examining Authority transmits herewith the international preliminary examination report and its annexes, if any, established on the international application.

2. A copy of the report and its annexes, if any, is being transmitted to the International Bureau for communication to all the elected Offices.

3. Where required by any of the elected Offices, the International Bureau will prepare an English translation of the report (but not of any annexes) and will transmit such translation to those Offices.

4. REMINDER

The applicant must enter the national phase before each elected Office by performing certain acts (filing translations and paying national fees) within 30 months from the priority date (or later in some Offices) (Article 39(1)) (see also the reminder sent by the International Bureau with Form PCT/IB/301).

where a translation of the international application must be furnished to an elected Office, that translation must contain a translation of any annexes to the international preliminary examination report. It is the applicant's responsibility to prepare and furnish such translation directly to each elected Office concerned.

For further details on the applicable time limits and requirements of the elected Offices, see Volume II of the PCT Applicant's Guide.

Name and mailing address of the IPEA/
Patent- och registreringsverket
Box 5055
S-102 42 STOCKHOLM
Facsimile No. 08-667 72 88

Telex
17978
PATOREG-S

Authorized officer

Madeline Cluskey

Telephone No. 08-782 25 00

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Hilary M McNulty
By: Hilary M. McNULTY

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(71) Applicant (for all designated States except US): **STERIS EUROPE INC. SUOMEN SIVULIIKE [FI/FI];** Teollisuustie 2, FIN-04300 Tuusula (FI).

(72) Inventors; and

(75) Inventors/Applicants (for US only): **NURMINEN, Teppo [FI/FI];** Uurrekuja 35, FIN-01650 Vantaa (FI). **STILL, Martin [FI/FI];** Haukantie 9 E 37, FIN-04320 Tuusula (FI).

(74) Agent: **OY JALO ANT-WUORINEN AB;** Iso Roobertinkatu 4-6 A, FIN-00120 Helsinki (FI).

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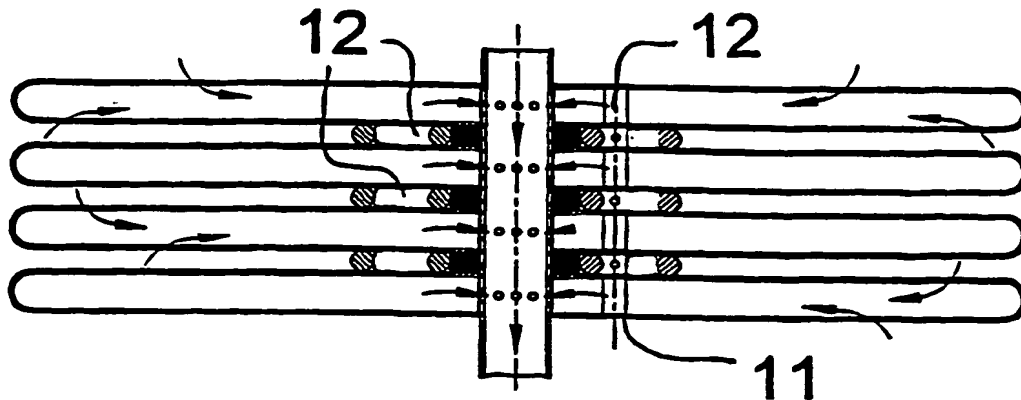
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For two-letter codes and other abbreviations, refer to the "Guidance Notes on Codes and Abbreviations" appearing at the beginning of each regular issue of the PCT Gazette.

(54) Title: **FILTERING UNIT AND METHOD OF SEALING SAME**



(57) Abstract: A crossflow filter comprising at least one membrane coated filtering units in a filter housing. The joints between the housing and the filtering unit are sealed by means of a gasket or a gasket system comprising an intermediate space. As the intermediate space is connected to the outside, a seal leak can be observed from emerging liquid. Thus, a substrate to be filtered cannot penetrate to the filtrate side as a consequence of a seal failure.

WO 01/03813 A1

FILTERING UNIT AND METHOD OF SEALING SAME

Field of the inventionField of the inventionField of the invention

The invention relates to filtration by means of crossflow technique and filtering elements having a filter membrane on a porous carrier.

Particularly, the invention relates to a crossflow membrane filtering unit having a sealing system including a channel enabling the detection of a leak without the quality of the filtrate being impaired.

Background of the inventionBackground of the inventionBackground of the invention

The removal of solids from a liquid by filtration can take place in several, principally different manners. In so-called dead end filtration, a suspension is driven in its entirety towards a filter, and a filter cake is formed which the filtrate penetrates. In cross-flow filtration, the mixture to be filtered is pumped across the filter element in a circuit, whereby part of the mixture penetrates the filtering elements forming a permeate stream. In the ideal case, no cake is formed, but the material to be separated is enriched in the circulating phase, and a certain fraction is continuously being removed therefrom as a reject stream. The quality of the permeate is basically dependant on the pore size of the filtering element membrane. This pore size can be very narrowly defined. In microfiltration, the pore diameter can be 0.1 - 10 μm , and in ultrafiltration down to 1 nm. Thus, separation of molecules on the basis of size is possible by means of filtration. One application is the removal of pyrogens from water, pyrogens having relatively large molecules.

Membrane filtration elements have porous carrier structures, on the filtering surfaces of which membranes having well defined pore sizes have been formed.

Membranes with small pore sizes can be formed from e.g. polymer films. These are not, however, particularly resistant to mechanical and thermal stress, which is a drawback in sterilisation and cleaning. Ceramic membranes on porous, ceramic carrier phases have also been developed. Most ceramic materials resist heat in addition to being mechanically durable. On a ceramic element, as on other materials, surfaces can also be sealed, i.e. made impermeable. Thus, three types of surfaces may occur on a ceramic filter element: membrane coated, sealed, and untreated, through which permeate flows freely.

For example, in US patent 5,104,546 a multilayer ceramic tubular filter element is disclosed, suitable for pyrogen removal by ultrafiltration. The end walls of the element as well as the inner walls of the channels are membrane coated. When the element is fitted into a shell or housing, and a mixture is pumped through the inner channels, permeate
5 flows through the porous carrier to the shell side. Seals, for example rubber gaskets, must be provided between the shell and the element end walls.

In addition to tubular elements, plate-shaped ceramic elements are generally known, see for example WO 98/28060 and references therein. A plate-shaped filter element has a
10 membrane coated outer surface and an aperture in the centre or elsewhere, the walls of which aperture are not membrane coated. Filters can be assembled by stacking such plates, as shown in e.g. GB 1 268 875. Due to the shape of the plates, the mating surfaces may be higher than the filter surfaces, whereby free filter area is provided between the plates as stacks are formed; plates may be separated by spacers; or if the seals between plates are
15 thick enough, the seals function as spacers. Thus, the central apertures form a permeate retrieval and exit channel, and the plate stack forms a flanged-tube structure across which the pressurised mixture to be filtered is allowed to flow. The mixture thus flows on the shell side and the permeate is collected from the inner channel.

20 As stacks are assembled, seals must be used between the plates and/or the spacers, and between the elements and the housing, the number of seals being proportional to the number of plates used.

Figure 1 shows a ceramic filter plate 1 according to the prior art, having a central bore 2.

25 Figure 2 shows a section of part of a stack formed from plates 1. Three plates are shown in the figure, but the stack may comprise additional plates depending on required filter surface area; the stack is confined in a housing not shown in the figure. In the housing, a flow of pressurised mixture for filtration is maintained. The plates comprise a porous ceramic support body 3, formed from e.g. aluminium oxide and having a relatively large pore size,
30 e.g. $> 1 \mu\text{m}$, and on the surface of the support body has been formed a ceramic filter membrane 4. The membrane has been formed from e.g. titanium or zirconium oxide. The central bores 2 of the plates form a permeate channel 6; in the embodiment shown, a perfo-

rated tube 5 is provided therein. The joints between the mating faces of the plates are sealed by means of O-rings 7.

During the filtration process, bodies smaller than the pores of membrane 4, as e.g. water molecules in the case of ultrafiltration, pass through the membrane due to the pressure difference, and are carried through the porous support body into the permeate chamber and exit through the end fittings.

In the above described filter units, the seals between the mixture and the permeate side are potential leak sites, and in demanding filtering processes a small leak may immediately render the permeate useless, as the mixture to be filtered penetrates into the permeate channel.

In tube or tube bundle type ceramic filter units, the sealing problem is associated with the end sections of the units. If the feed mixture penetrates the joint between the housing and the filter element and enters the shell side, the permeate is contaminated. In plate stack-type filter units, potential leak sites are the joints between the plates and between the plate stack and the housing.

Disclosure of the invention

A main object of the invention is a crossflow-type membrane filtering unit according to claim 1, wherein the sealing system between the filtering element and its housing comprises a channel through which the feed mixture on penetrating the seal system can flow without penetrating the whole sealing system, thereby indicating a leak.

In an embodiment using plate-shaped filtering elements, two concentric seals are preferably used between the filter plates. Thus, a space delimited by the seals and two plates is formed. At the location of said space, the plate surface is sealed, that is made impermeable. In addition, at the location of said space, at least one opening is provided in each plate, interconnecting the spaces between adjacent plates in the stack. The walls of the opening(s) are also sealed, and consequently an isolated, sealed space surrounding the permeate channel is formed. A tube or equivalent can be provided to connect this space to the outside. If a leak occurs in a seal between two plates, the fluid may thus flow into the

isolated space and out. Thus, a leaking seal can be detected immediately, and no possibility exists for the feed mixture to enter the permeate channel.

Preferably, the double seal system consists of two concentric, annular seals of different diameter. The seals may be of the O-ring type, and corresponding grooves may be provided in the filter plates. A huge range of O-ring seals designed for various chemical and physical conditions is available, and as the chemical and thermal durability of ceramic filter plates is usually high, a filter structure is thus provided which is functional in harsh conditions, with a low risk for permeate contamination by feed mixture.

In embodiments comprising filter elements of a tube or tube bundle structure, the joint between the filter housing and the filter element must be reliably sealed. The seal can be formed between the element end wall and the housing, or between the side walls of the element and the housing, and appropriate surface sealing of the filter element at the site of the seal system must be provided.

Surface sealing can be effected in various manners known to the person skilled in the art. On ceramic filter elements the surface may be glazed, or organic coatings may be used according to the process requirements. Organic coatings are obviously useful in the case of organic filtering elements. Metal foils is another possibility.

A further object of the invention is to provide a method of sealing a crossflow membrane filtering unit comprising one or more filtering elements within a housing, according to claim 6.

Preferably, filtering units according to the invention are used in water filtration, more preferably water ultrafiltration; most preferably filtering units according to the invention are used in the production of heat sterilised, pyrogen-free water.

Brief description of the drawings

Figure 1 shows a ceramic filter plate according to the prior art; figure 2 shows a sideways section of a plate stack according to the prior art, assembled from plates according to claim 1;

figure 3 is a schematic overview of a crossflow filtration system;

figure 4 shows a ceramic filter plate according to the present invention;

figure 5 shows a sideways section of a plate stack according to the present invention, assembled from plates according to claim 4;

5 figure 6 shows an embodiment of the present invention, wherein a tubular type filter element is used and the seal is located between the end wall of the element and the head of the housing;

figure 7 shows an embodiment of the present invention, wherein a tubular type filter element is used and the seal is located between the side walls of the element and the
10 housing.

Detailed descriptionDetailed descriptionDetailed description

The invention is described below with reference to the enclosed drawings.

In Figure 3, an overview of crossflow filtering system is shown. Material to be filtered
15 enters through the feed line 5, by means of pump 1. Pump 2 serves the filtration circuit, in which the mixture is pumped across filter unit 3. Filter unit 3 can be of any suitable type, e.g. the plate stack type or the tube or tube bundle type mentioned above. Part of the circulating fluid penetrates the pores of the filtering elements, forming the permeate, which leaves through line 6 or alternatively through reject line 7. To keep the concentration of
20 retained material in the filtration circuit at a desired level, a retentate stream is withdrawn through line 8, while the balance remains in the filtration circuit 9.

Figure 4 shows a filter plate according to the invention in a view corresponding to that of the prior art plate shown in Figure 1. In the central zone of the plate, a sealed-surface area 8
25 has been formed. In this area, two concentric O-ring seals 9, 10 of different diameters have been fitted, and in addition an opening 11 with sealed surfaces has been formed. Figure 5 shows a section analogous to fig. 2. Through openings 11, an essentially pressureless unitary space 12 is formed, surrounding the permeate channel and limited by the seal surfaces and sealed surface areas of the plates. The space 12 communicates with the
30 outside. Permeate that has penetrated the membrane into the porous carrier body cannot penetrate the sealed surface into space 12, but can only enter the permeate channel, as the walls of the central bores are not sealed. If a leak occurs in seal 9, the liquid leaking

through flows into space 12 and out, indicating the presence of a leak. As the pressure in the permeate channel is also greater than in space 12, also leaks occurring in the inner seals 10 are revealed, but the feed mixture is fully prevented from entering the permeate channel. Space 12 is connected to the outside e.g. via a tube through the filter housing.

The plates shown are circular, but other shapes may be used. For example, segments 13, 14 may be cut off as shown in Figures 1 and 5.

Figure 6 shows a partial section through the end of another type of filter unit, along a plane parallel to the filtration circuit flow. This unit comprises a tubular filter element 15, a housing head 16 and a seal 17 between these. The end wall of the filter element is sealed by the impermeable layer 24. The feed mixture enters at connection 18 and flows under pressure through the inner channels 20 of the element 15, coated with membrane 19. The permeate flows through the membrane and the porous body 25 of element 15 into the shell space formed by housing wall 21. According to the invention, seal 17 comprises an internal leak detection channel system 22, formed by grooves in the upper and lower surfaces of seal 17, said grooves being interconnected by evenly spaced openings. The channel system 22 communicates with the outside by channel 23, formed in housing head 16. As the feed mixture penetrates the inner part of the filter from the pressure side (18,20) of seal 17, it flows into channel 22 and out via channel 23, indicating the presence of a leak, regardless of whether the leak flow occurs along the upper or the lower face of the seal. The permeate side being normally under a higher pressure than the surroundings, permeate-side seal failures are likewise revealed when the leaking fluid seeps out of channel 23.

In the embodiment of Figure 7, seal 29 is provided between the end wall of tubular filter element 26 and the housing wall 27. The end surface 28 of the filter element and the wall surface section 30 at the site of filter element are impermeably sealed. On the inner face of seal 29 are provided groove 31 and at least one exit channel 32 connected to the outside, providing a leak indicating channel system in the same manner as illustrated in Figure 6. Possible leaks lead to flows from the joint between the housing head and the housing wall.

Automatic detection of leak flow may be provided in addition to or instead of visual observation. For example, moisture sensors may be provided in the intermediate spaces 12, 22, 31 or their corresponding exit channels, as shown schematically as 4 in Figure 3.

Claims

1. Crossflow ceramic membrane filtering unit having within a housing one or more filtering elements partly coated with a filtering membrane, **characterised** by the joints
5 between the filtering elements and between the filtering elements and the housing being sealed by means of a gasket or a gasket system comprising an internal intermediate space.

2. Membrane filtering unit according to claim 1, **characterised** by the intermediate space being connected to the outside of the filtering unit.

10 3. Membrane filtering unit according to claim 1 or 2, **characterised** by having a sensor indicating fluid leaking into the intermediate space.

15 4. Ceramic membrane filtering unit according to any claim 1-3, comprising one or more plate-shaped filtering elements having apertures, said elements being joined together and to the housing, said apertures thereby forming a tight, unitary conduit for permeate removal and the outer surface of the elements being essentially free to receive material to be filtered, **characterised** by the plate-shaped elements being fitted to each other and to the housing by means of at least two gaskets, and the central zones of the elements being
20 impermeable so as to form spaces delimited by the gaskets and the impermeable surfaces, and said spaces being interconnected by apertures in the elements, said apertures having impermeable surfaces.

25 5. Ceramic membrane filtering unit according to any claim 1-3, comprising at least one tubular filtering element having at least one channel internally coated with a filtering membrane.

30 6. Method for sealing a ceramic crossflow membrane filtering unit comprising one or more filtering elements within a housing, said elements being partly coated with a filtering membrane, **characterised** by sealing the joints between the elements and between the elements and the housing by a gasket or a gasket system comprising an internal space connected to the outside.

7. The use of a filtering unit according to claim 1 or a method according to claim 8 in the filtration, preferably ultrafiltration, of water.

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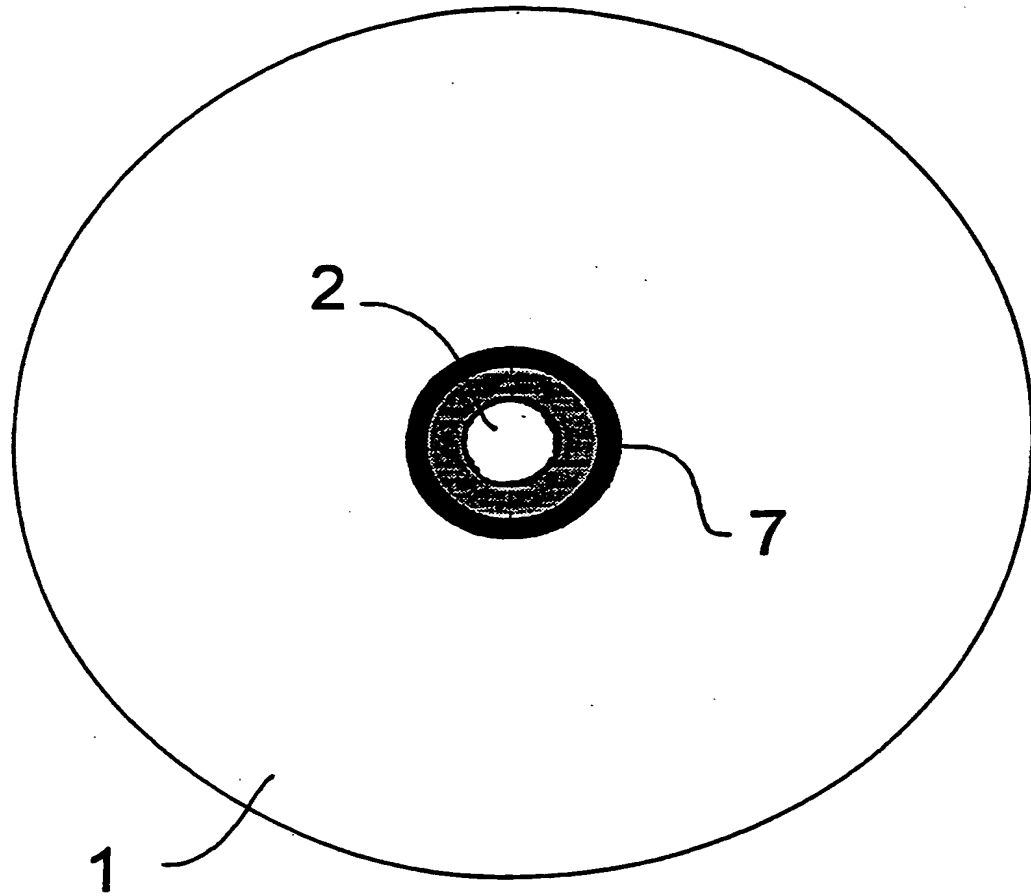


Fig. 1

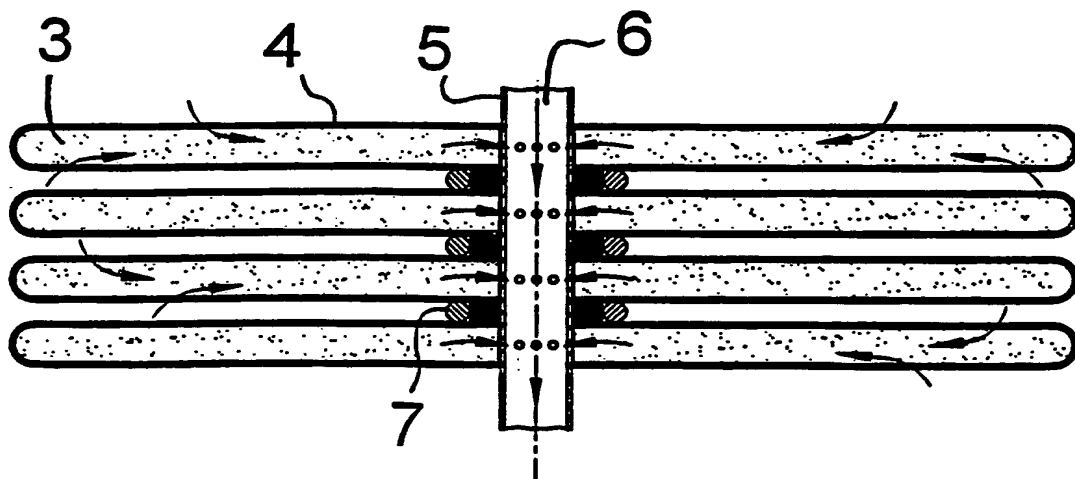


Fig. 2

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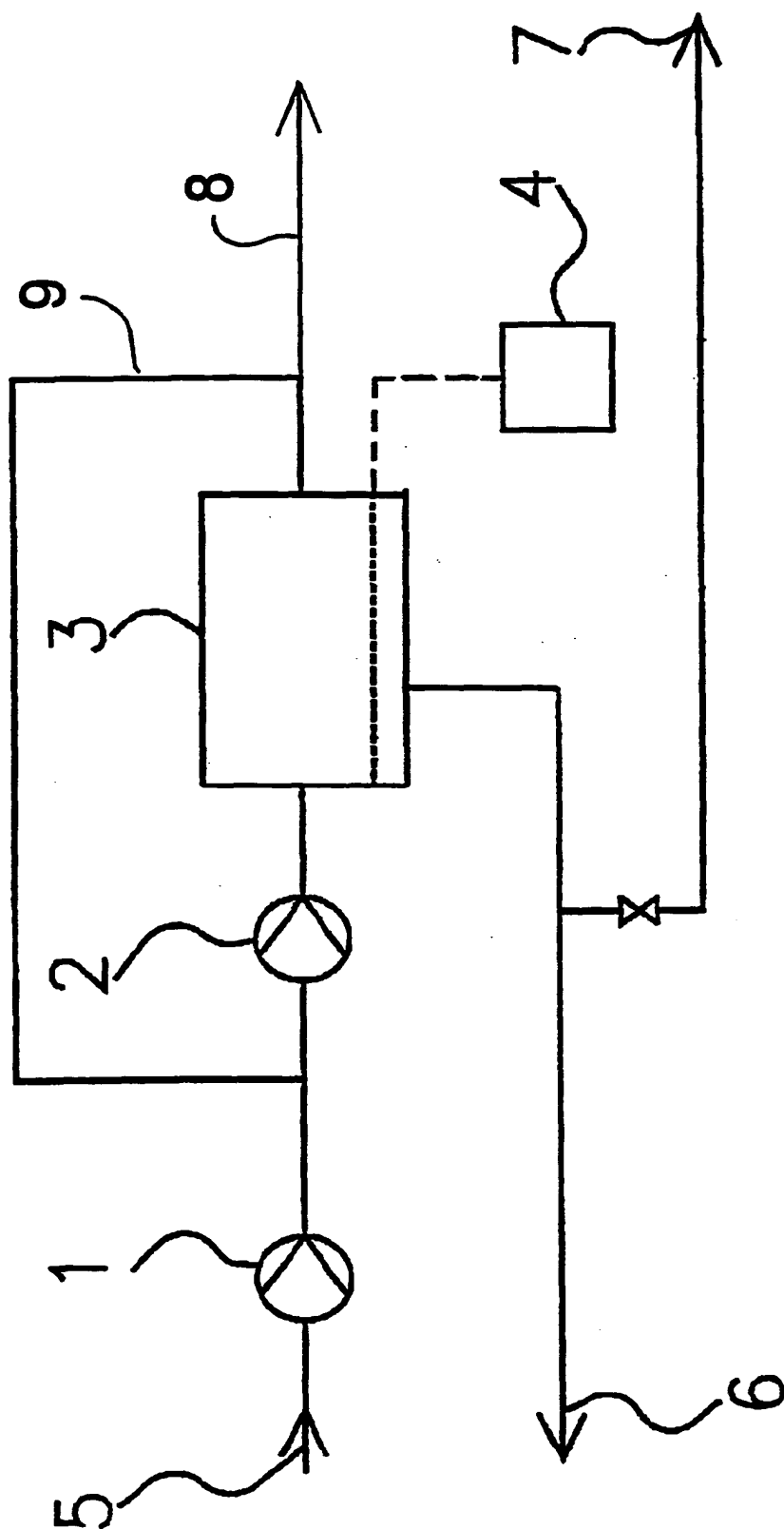


Fig. 3

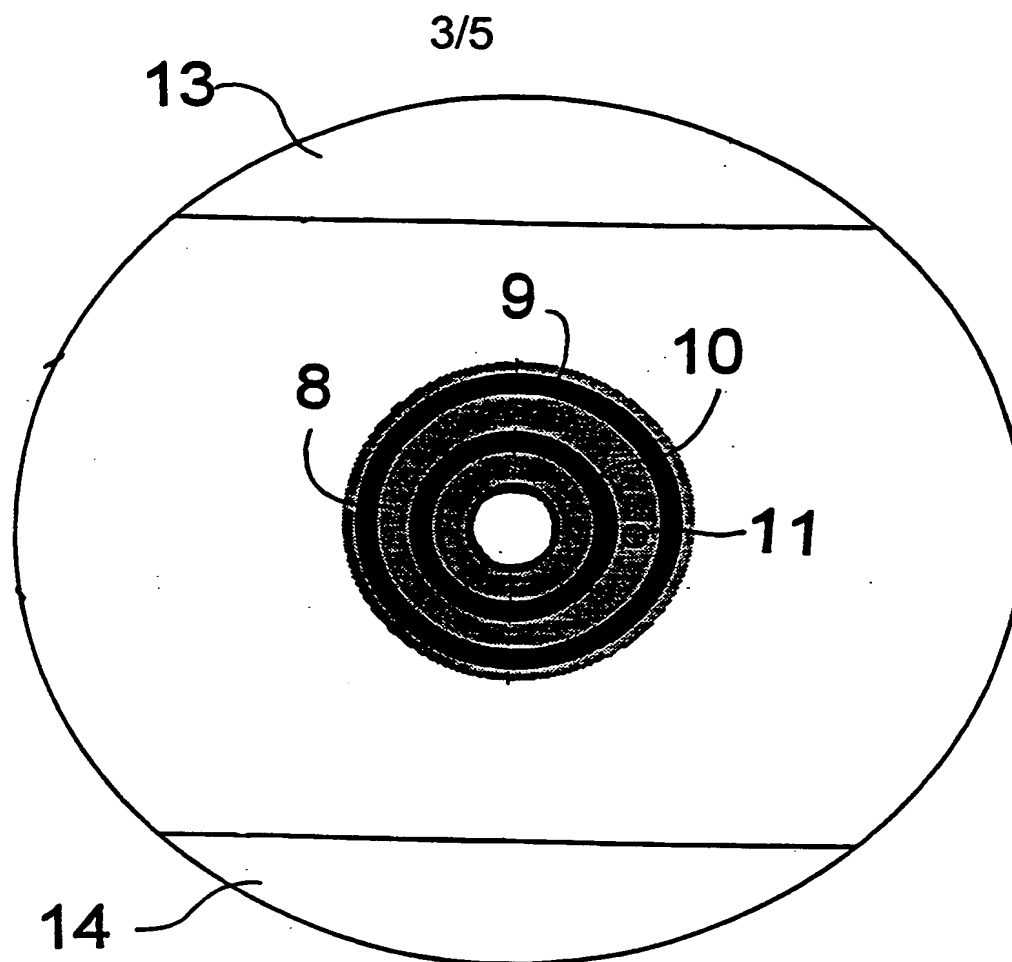


Fig. 4

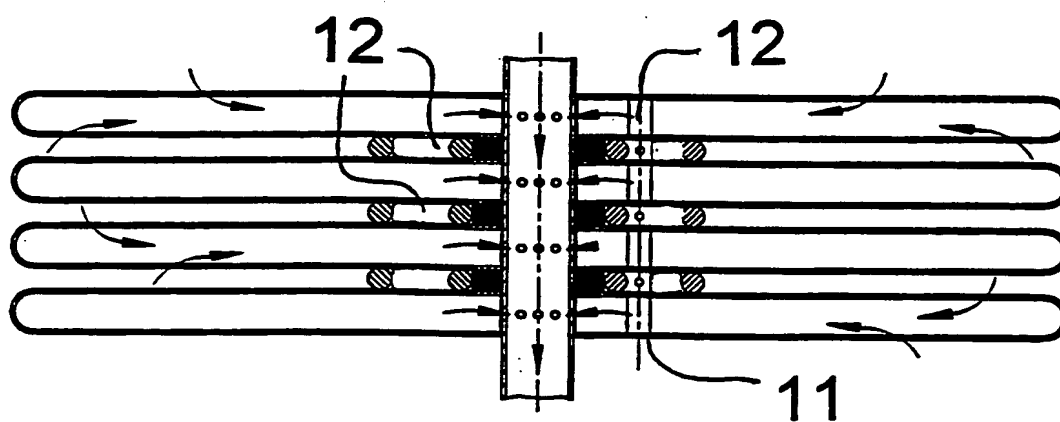


Fig. 5

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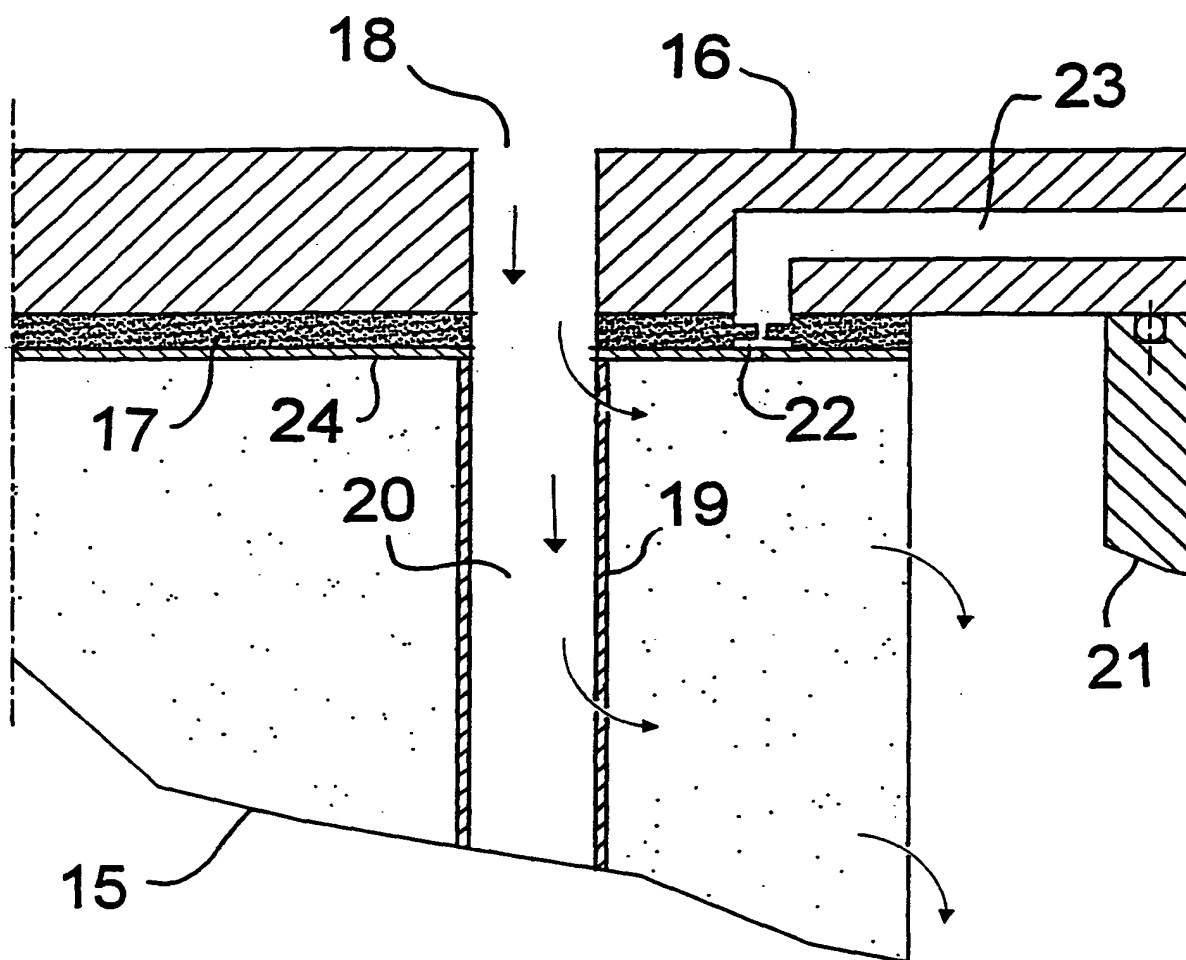


Fig. 6

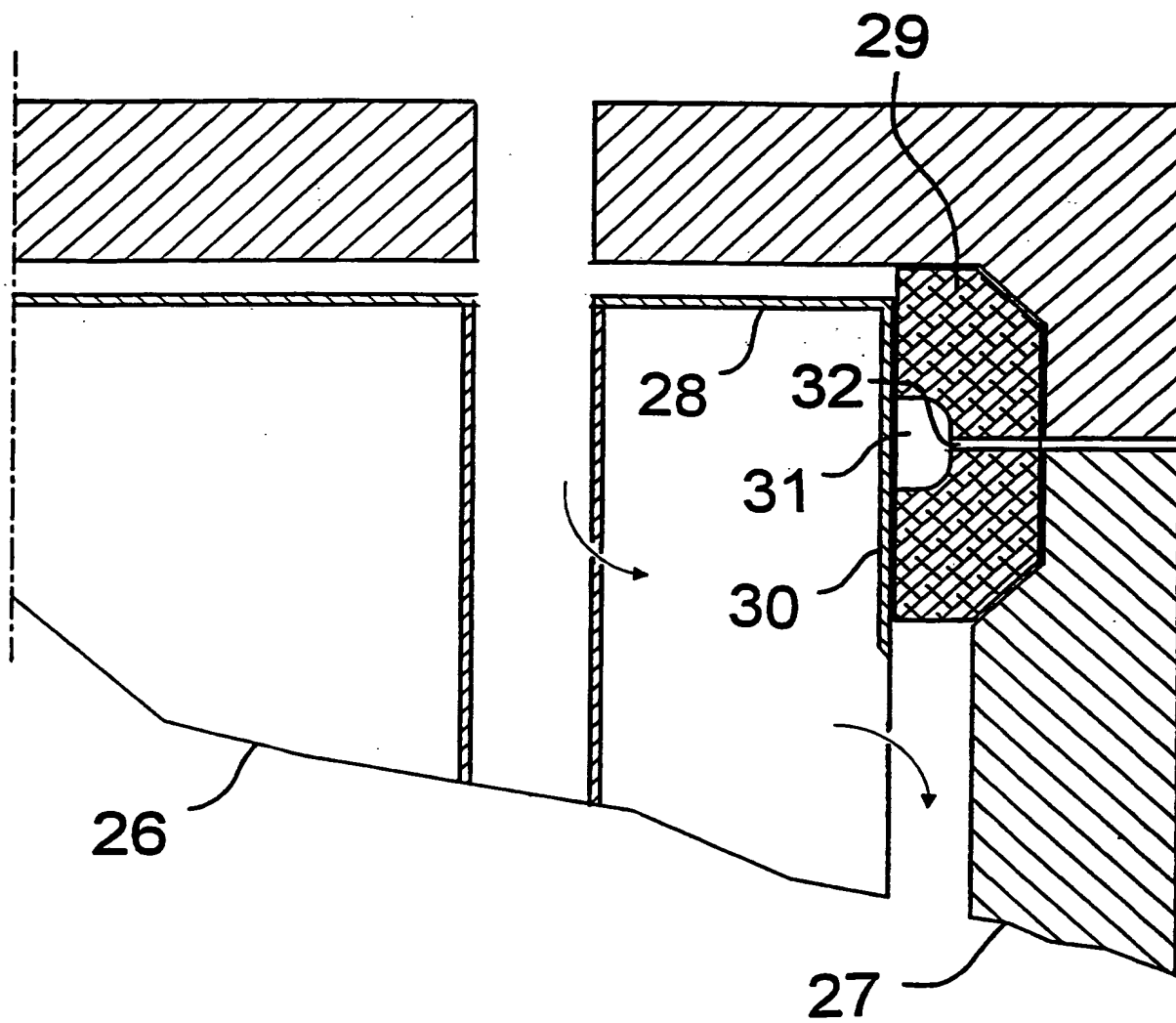


Fig. 7

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INTERNATIONAL SEARCH REPORT

International application No.
PCT/FI 00/00556

A. CLASSIFICATION OF SUBJECT MATTER

IPC7: B01D 65/00, B01D 61/14

According to International Patent Classification (IPC) or to both national classification and IPC

B. FIELDS SEARCHED

Minimum documentation searched (classification system followed by classification symbols)

IPC7: B01D

Documentation searched other than minimum documentation to the extent that such documents are included in the fields searched

SE,DK,FI,NO classes as above

Electronic data base consulted during the international search (name of data base and, where practicable, search terms used)

C. DOCUMENTS CONSIDERED TO BE RELEVANT

| Category* | Citation of document, with indication, where appropriate, of the relevant passages | Relevant to claim No. |
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| A | US 5015316 A (OSTREICHER ET AL), 14 May 1991 (14.05.91), abstract | 1-7 |
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☒ Further documents are listed in the continuation of Box C.

☒ See patent family annex.

* Special categories of cited documents:

"A" document defining the general state of the art which is not considered to be of particular relevance

"E" earlier application or patent but published on or after the international filing date

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"O" document referring to an oral disclosure, use, exhibition or other means

"P" document published prior to the international filing date but later than the priority date claimed

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"X" document of particular relevance: the claimed invention cannot be considered novel or cannot be considered to involve an inventive step when the document is taken alone

"Y" document of particular relevance: the claimed invention cannot be considered to involve an inventive step when the document is combined with one or more other such documents, such combination being obvious to a person skilled in the art

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Date of the actual completion of the international search

Date of mailing of the international search report

13 November 2000

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Box 5055, S-102 42 STOCKHOLM

Facsimile No. +46 8 666 02 86

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INTERNATIONAL SEARCH REPORT

International application No.

PCT/FI 00/00556

C (Continuation). DOCUMENTS CONSIDERED TO BE RELEVANT

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INTERNATIONAL SEARCH REPORT

Information on patent family members

02/11/00

International application No.

PCT/FI 00/00556

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INTERNATIONAL SEARCH REPORT

Information on patent family members

02/11/00

International application No.

PCT/FI 00/00556

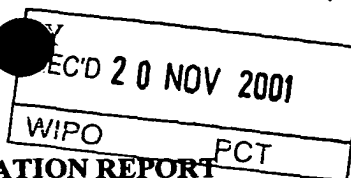
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PATENT COOPERATION TREATY

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INTERNATIONAL PRELIMINARY EXAMINATION REPORT

(PCT Article 36 and Rule 70)



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|---|---|--|
| Applicant's or agent's file reference 34695 | FOR FURTHER ACTION See Notification of Transmittal of International Preliminary Examination Report (Form PCT/IPEA/416) | |
| International application No. PCT/FI00/00556 | International filing date (day/month/year) 21.06.2000 | Priority date (day/month/year) 09.07.1999 |
| International Patent Classification (IPC) or national classification and IPC ₇ B 01 D 65/00, B 01 D 61/14 | | |
| Applicant Steris Europe Inc. Suomen Sivuliike et al | | |

1. This international preliminary examination report has been prepared by this International Preliminary Examining Authority and is transmitted to the applicant according to Article 36.

2. This REPORT consists of a total of 3 sheets, including this cover sheet.

☐ This report is also accompanied by ANNEXES, i.e., sheets of the description, claims and/or drawings which have been amended and are the basis for this report and/or sheets containing rectifications made before this Authority (see Rule 70.16 and Section 607 of the Administrative Instructions under the PCT).

These annexes consist of a total of _____ sheets.

3. This report contains indications relating to the following items:

- I ☒ Basis of the report
- II ☐ Priority
- III ☐ Non-establishment of opinion with regard to novelty, inventive step and industrial applicability
- IV ☐ Lack of unity of invention
- V ☒ Reasoned statement under Article 35(2) with regard to novelty, inventive step or industrial applicability; citations and explanations supporting such statement
- VI ☐ Certain documents cited
- VII ☐ Certain defects in the international application
- VIII ☐ Certain observations on the international application

| | |
|--|--|
| Date of submission of the demand 29.01.2001 | Date of completion of this report 13.11.2001 |
| Name and mailing address of the IPEA/SE Patent- och registreringsverket Box 5055 S-102 42 STOCKHOLM Facsimile No. 08-667 72 88 | Authorized officer Jan Carlerud/ELY Telephone No. 08-782 25 00 |

INTERNATIONAL PRELIMINARY EXAMINATION REPORT

International application No.

PCT/FI00/00556

I. Basis of the report

1. With regard to the elements of the international application:*

- ☒ the international application as originally filed
- ☐ the description:
 pages _____, as originally filed
 pages _____, filed with the demand
 pages _____, filed with the letter of _____
- ☐ the claims:
 pages _____, as originally filed
 pages _____, as amended (together with any statement) under article 19
 pages _____, filed with the demand
 pages _____, filed with the letter of _____
- ☐ the drawings:
 pages _____, as originally filed
 pages _____, filed with the demand
 pages _____, filed with the letter of _____
- ☐ the sequence listing part of the description:
 pages _____, as originally filed
 pages _____, filed with the demand
 pages _____, filed with the letter of _____

2. With regard to the language, all the elements marked above were available or furnished to this Authority in the language in which the international application was filed, unless otherwise indicated under this item.

These elements were available or furnished to this Authority in the following language _____ which is:

- ☐ the language of a translation furnished for the purposes of international search (under Rule 23.1(b)).
- ☐ the language of publication of the international application (under Rule 48.3(b)).
- ☐ the language of the translation furnished for the purposes of international preliminary examination (under Rules 55.2 and/or 55.3).

3. With regard to any nucleotide and/or amino acid sequence disclosed in the international application, the international preliminary examination was carried out on the basis of the sequence listing:

- ☐ contained in the international application in written form.
- ☐ filed together with the international application in computer readable form.
- ☐ furnished subsequently to this Authority in written form.
- ☐ furnished subsequently to this Authority in computer readable form.
- ☐ The statement that the subsequently furnished written sequence listing does not go beyond the disclosure in the international application as filed has been furnished.
- ☐ The statement that the information recorded in computer readable form is identical to the written sequence listing has been furnished.

4. ☐ The amendments have resulted in the cancellation of:

- ☐ the description, pages _____
- ☐ the claims, Nos. _____
- ☐ the drawings, sheet/fig _____

5. ☐ This report has been established as if (some of) the amendments had not been made, since they have been considered to go beyond the disclosure as filed, as indicated in the Supplemental Box (Rule 70.2 (c)).**

* Replacement sheets which have been furnished to the receiving Office in response to an invitation under Article 14 are referred to in this report as "originally filed" and are annexed to this report since they do not contain amendments (Rules 70.16 and 70.17).

** Any replacement sheet containing such amendments must be referred to under item I and annexed to this report.

INTERNATIONAL PRELIMINARY EXAMINATION REPORT

International application No.

PCT/FI00/00556

V. Reasoned statement under Article 35(2) with regard to novelty, inventive step or industrial applicability; citations and explanations supporting such statement**1. Statement**

| | | | |
|-------------------------------|--------|------------|-----|
| Novelty (N) | Claims | <u>1-7</u> | YES |
| | Claims | | NO |
| Inventive step (IS) | Claims | <u>1-7</u> | YES |
| | Claims | | NO |
| Industrial applicability (IA) | Claims | <u>1-7</u> | YES |
| | Claims | | NO |

2. Citations and explanations (Rule 70.7)

Documents cited in the International Search Report:

- A) US 5422057 A
- B) GB 2135902 A
- C) US 5015346 A
- D) EP 0038612 A2

The present invention relates to a crossflow filter and is intended to provide a solution to the problem of detecting a leak without the quality of the filtrate being impaired.

Document B discloses a hollow fiber type filtering unit comprising gaskets for sealing the joint between the filtering elements and the housing. Between the gaskets there is an intermediate space connected to the outside of the filtering unit. A measuring apparatus can be provided to indicate a leak. (See p.3, lines 7-19, fig.4). However, there is no indication in any of the cited documents to apply the technique disclosed in B to known crossflow filter technique as exemplified by documents A, C and D.

Therefore, on reconsidering, the invention is novel and is considered to involve an inventive step and to be industrially applicable.

PATENT COOPERATION TREATY

SAAPUNUT

From the
INTERNATIONAL PRELIMINARY EXAMINING AUTHORITY

20. 11. 2001

PCT

Oy JALO ANT-WUORINEN A

To:

OY JALO ANT-WUORINEN AB
Iso Roobertinkatu 4-6 A
FIN-00120 Helsinki
Finland

NOTIFICATION OF TRANSMITTAL OF
INTERNATIONAL PRELIMINARY
EXAMINATION REPORT

(PCT Rule 71.1)

Date of mailing
(day/month/year)

16-11-2001

Applicant's or agent's file reference

302709

IMPORTANT NOTIFICATION

International application No.

PCT/FI00/00556

International filing date (day/month/year)

21-06-2000

Priority date (day/month/year)

09-07-1999

Applicant

Steris Europe inc. Suomen Sivuliike
et al

1. The applicant is hereby notified that this International Preliminary Examining Authority transmits herewith the international preliminary examination report and its annexes, if any, established on the international application.
2. A copy of the report and its annexes, if any, is being transmitted to the International Bureau for communication to all the elected Offices.
3. Where required by any of the elected Offices, the International Bureau will prepare an English translation of the report (but not of any annexes) and will transmit such translation to those Offices.
4. REMINDER

The applicant must enter the national phase before each elected Office by performing certain acts (filing translations and paying national fees) within 30 months from the priority date (or later in some Offices) (Article 39(1)) (see also the reminder sent by the International Bureau with Form PCT/IB/301).

where a translation of the international application must be furnished to an elected Office, that translation must contain a translation of any annexes to the international preliminary examination report. It is the applicant's responsibility to prepare and furnish such translation directly to each elected Office concerned.

For further details on the applicable time limits and requirements of the elected Offices, see Volume II of the PCT Applicant's Guide.

Name and mailing address of the IPEA/

Patent- och registreringsverket
Box 5055
S-102 42 STOCKHOLM
Facsimile No. 08-667 72 88

Telex
17978
PATOREG-S

Authorized officer

Madelon Clabon

Telephone No. 08-782 25 00